

LABORATORY ANALYSIS FACT SHEET

TPH-GRO by Method 8260B

The Missouri DNR has adopted SW-846 method 8260B as the method of choice for the detection and quantitation of TPH-GRO. The following items represent exceptions or clarifications to the method as written.

Sample Introduction

1. Samples are purged onto the GC/MS system using all protocols specified in SW-846 method 5035 or 5035A.
2. Surrogates and internal standards specified by method 8260B are added to water and soil samples prior to purging. Since the surrogate Dibromofluoromethane is not related to compounds detected by this method, it does not have to be reported nor does it have to meet any acceptance criteria.

Sample Analysis

1. The GC/MS system is tuned to BFB tune criteria listed in method 8260B, at the frequency specified in method 8260B. Alternative tuning criteria as specified in section 7.3 of method 8260B is also acceptable, as long as method performance is not adversely affected.
2. The GC/MS operating conditions specified as guidance in Section 7.3 of Method 8260B are acceptable.
3. A 5 point standard curve is used to quantitate TPH-GRO. Quantitation is by external standard.
4. The stock standard solution used to prepare calibration standards is a mixture of unleaded gasoline and Number 2 diesel fuel. The stock solution and all calibration standards use Methanol as the solvent. Each component should be at a concentration of 1000 mg/L in the stock standard.
5. The lowest calibration standard should be at the reporting limit for the method. The highest calibration standard must not exceed the linear range of the system.
6. Retention time windows are defined for TPH-GRO by analyzing a standard containing C6 and C10. The retention time window for TPH-GRO is defined as beginning 0.1 minutes before C6 to 0.1 minutes after C10. The standard containing C6 and C10 must be analyzed every day samples are analyzed in order to verify that the retention time windows are constant.
7. Because the retention time window will be several minutes wide for TPH-GRO, the GC/MS data system may not accurately or appropriately establish the proper baseline

for calibration or quantitation. The analyst **must** visually examine the computer-generated baseline for every analytical run, and manually adjust the baseline when needed. A properly drawn baseline must extend over the entire retention time window and include the area under the entire TPH-GRO series of peaks. It is **not appropriate** to draw the baseline “peak to peak.”

8. Individual quantitation ions are not appropriate for TPH-GRO calibration and quantitation. The Total Ion Chromatogram (TIC) must be used to calculate the area under the peak for TPH-GRO calibration and quantitation determinations over the entire retention time window.
9. Area counts for the internal standards and surrogates added during sample preparation must be subtracted from the total area count for TPH-GRO. This is accomplished by subtracting the area count of the method blank from all subsequent calibration and analytical runs.
10. The %RSD for the calibration curve for TPH-GRO must be less than or equal to 20 percent, so that linearity through the origin can be assumed and an average calibration factor used for calculations.
11. A continuing calibration check verification standard (CCV) must be analyzed every 12 hours of sample analysis. The CCV must contain all analytes reported by this method. The standard concentration should be at the mid-point of the calibration curve. If the percent difference (%D) for the CCV is less than or equal to 20%, the initial calibration is assumed to be valid. If the %D is greater than 20%, corrective action must be taken prior to sample analysis.
12. A method blank must be analyzed for every 20 samples analyzed. Additionally minimum quality control samples per every 20 samples include a Laboratory Control Sample (LCS) and Matrix Spike/Matrix Spike Duplicate (MS/MSD). Ideally, the spiking solution(s) for the LCS and MS/MSD would contain TPH-GRO, BETX, chlorinated compounds, and oxygenates. It may not be possible however to have every component in the spiking solution(s) for the LCS and MS/MSD samples. It is acceptable to spike the LCS and MS/MSD with different components, as long as TPH-GRO, BETX, chlorinated compounds, and oxygenates are present in either the LCS or the MS/MSD samples.
13. All of the BTEX, chlorinated compounds and oxygenates analyzed by 8260B are by internal standard, using all criteria specified in 8260B. This includes the RSD requirement of 15% for the compounds. If 15% RSD is not achieved, additional calibration options specified in Method 8000B may be employed.

Should questions arise concerning TPH analysis by Method 8270C, feel free to contact either of the following people:

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